ENVIRONMENTAL RADIOACTIVITY LABORATORY INTERCOMPARISON STUDIES PROGRAM 1973 - 1974

by

Quality Assurance Branch Technical Support Laboratory National Environmental Research Center Las Vegas, Nevada

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NATIONAL ENVIRONMENTAL RESEARCH CENTER OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY LAS VEGAS, NEVADA 89114

PREFACE

Quality assurance is an integral part of any viable environmental monitoring activity. The primary goals of the Environmental Protection Agency's (EPA) quality assurance program are to improve and document the credibility of environmental measurements. To achieve these goals, quality assurance is needed in nearly all segments of monitoring activities and should cover personnel, methods selection, equipment, and data handling procedures.

Five major functions, each essential to an effective quality assurance effort, comprise EPA's quality assurance program:

- Promulgation of standardized methods of measurement
- Distribution of standard reference materials
- Issuance of guidelines and procedures
- Training and technical assistance
- Evaluation and certification of monitoring activities

This manual has been prepared to assist laboratories involved with environmental radiation measurements in developing and maintaining a quality control program and documenting the precision and accuracy of their data. All EPA monitoring programs are requested to make use of this document in planning their own radiation measurements and in assisting the States in carrying out radiation monitoring activities.

Comments concerning the utility of this document, along with any suggestions for possible changes and revisions, are welcomed. Questic on matters related to quality assurance of environmental measurements in various field should be directed to the following person(s):

Air Pollution

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Radiation

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During the months ahead, manuals and docoments will be issued which provide guidelines to be followed in all phases of monitoring activities--sampling, analysis, and data handling--and in all media; air, water, and land. Use of these guidelines throughout the Agency will enable a uniform approach to be established within EPA which ultimately can be implemented at the State level. This should permit a significant improvement in the validity and reliability of environmental data which the Nation collects.

The implementation of a total and meaningful national environmental quality assurance effort cannot succeed without the full support of all monitoring programs. Your cooperation is appreciated.

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I. THE LABORATORY INTERCOMPARISON STUDIES PROGRAM

Environmental measurements for radiation are made daily by many different Federal, State, local and private agencies. The data from these measurements are used for a wide variety of purposes, including health effects, the establishment of standards and guides, and for enforcement activities. It is therefore imperative that the precision and accuracy of the data be assured in order that policy decisions concerning environmental quality are based upon valid and comparable data.

In order to attain this goal, an Agency-wide quality assurance program has been implemented within the U.S. Environmental Protection Agency (EPA). In the area of radiation, quality control responsibilities have been assigned to the Quality Assurance Branch at the EPA's National Environmental Research Center-Las Vegas which carries out a program designed to encourage the development and implementation of quality control laboratory procedures for sample collection, analysis, data handling, and reporting purposes.

A major objective of this program is to assist laboratories involved in environmental radiation measurements to develop and maintain both an intralaboratory and an interlaboratory quality control program. In part, this is accomplished through an extensive laboratory intercomparison study ("cross-check") program involving environmental media (milk, water, air, food, soil and gases) and a variety of radionuclides with activities at or near environmental levels.

A number of different environmental samples, containing precisely known amounts of one or more radionuclides, are prepared and routinely distributed to all laboratories requesting them. These laboratories perform the required analyses and return their data to

the Quality Assurance Branch for statistical analysis and comparison with other participating laboratories. A computer report and a continually updated performance chart are returned to each participant. The program thus enables a laboratory to document the precision and accuracy of radiation data, identify instrumental and procedural problems, and to compare performance with other laboratories.

Each laboratory participating in a cross-check study is expected to have an internal quality control program in operation to insure that all instrumentation is calibrated and functioning and that analytical procedures are being carried out properly. Such a program includes continual monitoring of instrumentation, the plotting of instrument control charts, frequent analysis of replicate samples to check precision, and the regular measurement of samples to which known amounts of activity have been added, to check the accuracy of systems.

Participation in a laboratory intercomparison does not automatically guarantee the precision and accuracy of a laboratory's data and should not be considered as a substitute for a continuous quality control program within a laboratory. However, intercomparison data may be useful either for indicating previously unsuspected instrumental problems or procedural deficiencies, or for documenting the precision, accuracy and validity of a laboratory's work. Participation in a laboratory intercomparison study program thus serves as a check on the internal quality control program.

If your laboratory does not now participate in the cross-check activities of the Quality Assurance Branch at the NERC-LV, or if you wish to expand its participation, you are encouraged to do so.

II. TYPES OF ENVIRONMENTAL SAMPLES DISTRIBUTED

The current laboratory intercomparison studies program involves the analysis of a variety of media containing environmental, or near environmental, levels of radioactivity. These include:

MILK

Four-liter milk samples containing 40 K, 89 Sr, 90 Sr, 131 I, 137 Cs, and 140 Ba are distributed on a monthly basis.

WATER

Water containing several different mixtures of radioactive materials are included in the cross-check program.

- Four-liter samples for the analysis of gross alpha and gross beta analysis are sent to participating laboratories every other month.
- Four-liter samples containing ⁵¹Cr, ⁶⁹Zn, ⁶⁰Co, ¹⁰⁶Ru, ¹³⁴Cs, and ¹³⁷Cs for gamma analysis are distributed every other month.
- Sixty-milliliter samples for tritium analysis, are mailed on a monthly basis.
- Four-liter water samples containing ²³⁹Pu are shipped to laboratories during May and November of each year.
- Four-liter samples of well water containing ²²⁶Ra are distributed in November, January, May and July.

AIR

Air filters, with optional 2-inch or 4-inch diameters, are sent out on a quarterly basis for gross alpha, gross beta, and ²³⁹Pu analysis.

SOIL

One-hundred-gram soil samples, containing ²³⁹Pu, will be distributed twice each year beginning in December 1973.

F00D

Three 4-liter food slurries containing 89 Sr, 90 Sr, 131 I, 137 Cs, 140 Ba, and K are sent to participants once each quarter.

URINE

Urine samples (60 ml) containing tritium are shipped on a quarterly basis.

GASES

4

Cylinders containing either $^{85}{\rm Kr}$ or $^{133}{\rm Xe}$ are distributed twice each year.

Sample size, activity levels, type of analysis, and other pertinent information concerning the cross-check samples are summarized in Table 1. The distribution schedule is outlined in Table 2.

A laboratory may participate in as few or as many of the studies as it desires.

TABLE 1 SUMMARY OF CROSS-CHECK PROGRAMS*

SAMPLE	ANALYSIS	ACTIVITY PER ISOTOPE	QUANTITY SUPPLIED	PRESERVATIVE	DISTRIBUTION	TIME FOR ANALYSIS & REPORT
Milk	89Sr, 90Sr, 131I, 137Cs, 140Ba, K	< 200 pCi/l	~ 4 liters	Formalin	Monthly	6 weeks
Water						
Gross α, β*	Gross α,	< 100 pCi/l	~ 4 liters	.5 N HNO3	Bimonthly	4 weeks
Gamma	60Co, ¹⁰⁶ Ru, ¹³⁴ Cs, ¹³⁷ Cs, ⁵¹ Cr, ⁶⁵ Zn	< 500 pCi/l	~ 4 liters	.5 N HNO ₃	Bimonthly	4 weeks
9Н	НE	< 3500 pCi/l	~ 60 ml	none	Monthly	4 weeks
239 Pu *	239 pu	< 10 pCi/l	~ 4 liters	.5 N HNO ₃	Semianually	6 weeks
226 Ra	226 Ra	< 20 pCi/l	~ 4 liters	.5 N HNO ₃	Quarterly	6 weeks
Air						
Gross α, β*	Gross α, β	< 200 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	4 weeks
239Pu*	239Pu	< 2 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	6 weeks
Soil*	239 p u	< 50 pCi/sample	- 100 g	none	Semiannually	6 weeks
Diet	89Sr, 90Sr, 131I, 137Cs, 140Ba, K	< 200 pCi/kg	3 - 4-liter samples	Formalin	Quarterly	6 weeks
Urine	3н	< 3500 pCi/l	~ 60 ml	Formalin	Quarterly	4 weeks
Gas	⁸⁵ Kr, ¹³³ Xe	< 20 pCi/ml	10 liters	none	Semiannually	6 weeks

*Laboratories are required to have the necessary licenses before receiving these samples.

TABLE 2

CROSS-CHECK SAMPLE DISTRIBUTION SCHEDULE

Numbers 1, 2, 3 & 4 indicate week of the month.

				_			$\overline{}$								
Gas	¹³³ Xe, ⁸⁵ Kr				7							3			
Urine	Н _Е			2			2				2			2	
Diet	Sr, Y		2		4					4		4			
Soil	γ, ²³⁹ Pu						3							4	
ilter	239Pu			3					3						m
Air Filter	Gross a, B				3					3				3	
	226Ra					3			3				3		3
	239 Pu					2							2		
Water	Н _С		2	2	2	2	2		2	2	2	2	2	2	2
	>			4		4			4		4		4		4
	Gross a, B		3		3		3			3		3		3	
Milk	Sr, Y		1	1	1	1	-1		1	H	-	П	1		1
	Month	1973	Aug	Sep	Oct	Nov	Dec	1974	Jan	Feb	Mar	Apr	May	Jun	Jul

III. ANALYSIS OF DATA

Each participating laboratory is expected to carry out three independent determinations for each radionuclide included in a particular cross-check and to report its results on a form (Figure 1) provided with the sample.

Upon receipt of the reports from all participating laboratories, the data are transferred to punch cards and introduced into a CDC 6400 computer for analysis. As indicated in the sample calculations (Appendix I), this analysis includes determination of the experimental standard deviation, calculation of the normalized range, normalized deviation, experimental error, and the grand average of all laboratories. The expected analytical precision values used as a basis for judging laboratory performance for specific nuclides are summarized in Table 3.

A report is generated containing the data reported by all participating laboratories, listed according to their identity code, along with the results of the computer analysis (Table 4, Figure 2). In addition, a control chart is generated and reproduced for each radionuclide included in the sample (Figures 3 and 4). The control charts are updated each time a laboratory participates in a particular cross-check study, thus giving each laboratory a continuous record of its performance.

A copy of the computer printout and a control chart for each radionuclide is mailed to each participant approximately 2 weeks following the report due date.



U.S. ENVIRONMENTAL PROTECTION AGENCY NATIONAL ENVIRONMENTAL RESEARCH CENTER LAS VEGAS, NEVADA

QUALITY ASSURANCE BRANCH

Laboratory		ID	
Contact Person	Phone		
Sample Type	ritium in Water		···
Collection Date8	8-10-73 0400 PST		
Analysis # NOTE: All resu	#3 ults are in pCi/l with background ted. Total activity is less than		
U.S. Enviro National Er		3*	to:

*Results not received by this date will not be included in final report.

Figure 1. Results reporting form

Table 3

Laboratory Precision

One Standard Deviation Values

For Various Analyses

Nuclide	<u>Level</u>	Standard Deviation Single Determination
1311	5 to 100 pCi/l or Kg > 100 pCi/l or Kg	5 pCi/1 5%
¹⁴⁰ Ba	5 to 100 pCi/l or Kg > 100 pCi/l or Kg	5 pCi/l 5%
¹³⁷ Cs	5 to 100 pCi/l or Kg > 100 pCi/l or Kg	5 pCi/1 5%
⁸⁹ Sr	5 to 100 pCi/l or Kg > 100 pCi/l or Kg	5 pCi/l 5%
⁹⁰ Sr	2 to 30 pCi/1 or Kg > 30 pCi/1 or Kg	1.5 pCi/l 5%
K	\geq 0.1 g/1 or Kg	0.06 g/1
Ca	≥ 0.1 g/1 or Kg	0.02 g/1 or Kg
Gross Alpha	> 1 pCi/1	25%
Gross Beta	1 to 25 pCi/1 > 25 to 130 pCi/1 > 130	25% 6.5 pCi/1 5%
³ H	< 4000 pCi/1 > 4000 pCi/1	% 1s = 16985x(pCi/1)9067 10%
²²⁶ Ra	> 0.1 pCi/1	15%
239 P u	\geq 0.1 pCi/l, gram or sample	10%

<u>Title</u> :	Program name, sample collection date, sample code letter, analysis type, known concentration of radio-nuclide, expected standard deviation of analysis - single determination.
Column 1:	Laboratory identification code (A, B, C, etc.).
Column 2:	Laboratory results (0-25 results listed down column).
Column 3:	1 s (standard deviation) of the experimental results.
Columns 4 & 5:	Normalized Range Value in "Mean Range + Standard Error of the Range" $(\bar{R} + \sigma_R)$ units for comparability. (See Statistical Techniques for Quality Control of Environmental Radioassay, AQCS Report Stat-1, November 1964, pp 4-8) $(S_R = \sigma_R)$ for printing purposes).
Column 6:	Average Value.
Column 7:	Normalized Deviation from the Grand Average Value of all laboratories expressed in $\sigma_{\mbox{\scriptsize M}}$ units.
Column 8:	Normalized Deviation from the Known Value expressed in $\sigma_{\mbox{\scriptsize M}}$ units.
Bottom of Chart:	l s experimental error of all laboratories and the Grand Average of all laboratories.

Figure 2. Explanation of terms used in participant date report

Table 4. SAMPLE ANALYSIS AND REPORT OF PARTICIPANT'S DATA

NERC-LV TRITIL IN WATER CROSSCHECK PROGRA

		12/27/72	SAMPLE - A	3н		
		KNOWN-VALUE =	1579.00 +/-	332.00	PCI/L	
		EXPERTMENTAL	<u> </u>		NORMALIZED	DEVIATION
LAB	RESULT	SIGMA	₹ + R	AVERAGE	(GRAND-AVG)	(KNOWN)
ŋ	1500.0					
ŋ	1400.0					
Ö	1400.0	57.74	.18 + 0	1433.3	-1.49	76
P	1872.0					
Þ	1688.0					
Р	1596.0	140.53	•49 + 0	1718.7	00	•73
46	1830.0					
AG	1810.0					
AG	1740.0	47.26	.16 + 0	1793.3	•39	1.12
ΔН	1626.0					
AΉ	1477.0					
ΔН	1652.0	94.43	.31 + 0	1585.0	70	.03
ΔI	2011.0					
ΑŢ	1713.0					
A T	2473.0	382.94	1 + •41	2065.7	1.81	2.54
Ε	NO DAT	TA PROVIDED				

EXPERIMENTAL SIGMA (ALL LABS) = 272.2 GRAND AVERAGE = 1719.2

^{* -} NOT USED FOR CALCULATING GRAND AVERAGE ** - INSUFFICIENT INFORMATION TO CALCULATE

Title: Name of Program, Laboratory Code Letter, and Type

of Analysis.

Upper Graph: "Normalized Deviation from Known versus the Month

of Analysis." (The 95.0% (μ ± $2\sigma_{\mbox{\scriptsize M}})$ and the 99.7% $(\mu \pm 3\sigma_M)$ Confidence Levels were chosen as the Warning Levels and Control Limits respectively.)

Middle Graph: "Normalized Deviation from the Grand Average Value

of All Laboratories versus the Month of Analysis."

(The 95.0% (x \pm 2 $\sigma_{\rm M}$) and the 99.7% (x \pm 3 $\sigma_{\rm M}$)

Confidence Levels were chosen as the Warning Levels

and Control Limits respectively.)

"Normalized Range Values (\bar{R} + σ_R) versus the Month of Analysis." (The 97.5% (\bar{R} + $2\sigma_R$) and ~100% (\bar{R} + $3\sigma_R$) Lower Graph:

Confidence Levels were chosen as the Warning Levels

and Control Limits respectively.)

Explanation of terms used in the control chart

TRITIUM IN WATER CROSS CHECK PROGRAM

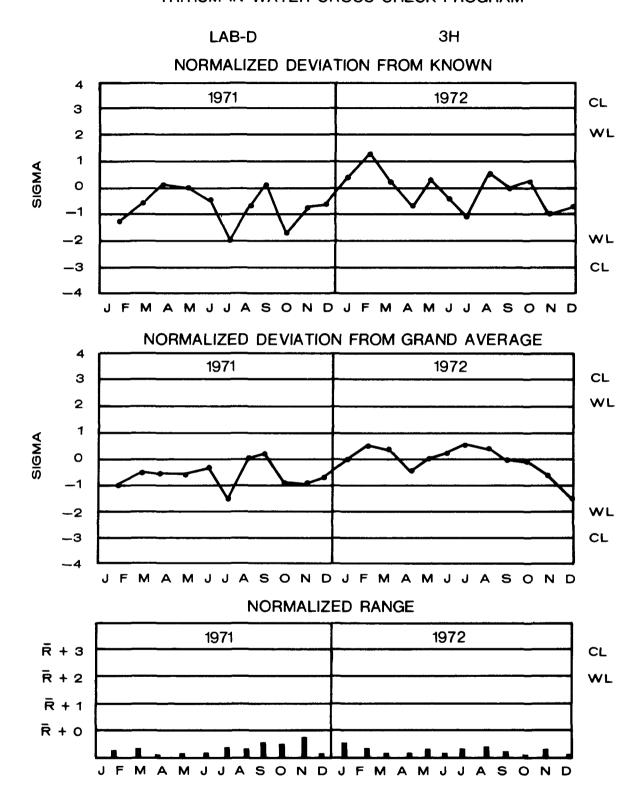


Figure 4. Control chart

IV. PARTICIPATION IN THE LABORATORY INTERCOMPARISON STUDIES PROGRAM

Any laboratory involved in, or concerned with, environmental radiation monitoring and surveillance, is eligible to participate in any one or all of the cross-checks described. Moreover, dependent upon personnel available and their workload, a laboratory may elect to receive samples on a less frequent basis than indicated on the distribution schedule (Table 2).

To become a participant in the laboratory intercomparison studies program, complete one of the forms included in this publication and return to:

U.S. Environmental Protection Agency National Environmental Research Center Quality Assurance Branch P.O. Box 15027 Las Vegas, NV 89114

Should the laboratory require additional types of cross-check samples at some later date, a second form--indicating the samples desired--should be submitted.

V. APPENDIX

Statistical Calculations

To illustrate the computations performed by computer, sample calculations are given using data for three hypothetical samples analyzed at one laboratory.

First, the analytical results are listed and the mean (sample calculation a) and the standard deviation (sample calculation b) are computed. These statistics provide measures of the central tendency and dispersion of the data. In our example the mean, \overline{X} , equals 60 pCi/l and the standard deviation, S, equals 10 pCi/l.

Next, the normalized range (sample calculation c) is computed by first finding the mean range, \overline{R} , the control limit, CL, and the standard error of the range, σ_R . The normalized range measures the dispersion of the data (precision) in such a form that control charts may be used. Control charts allow one to readily compare past analytical performance with present performance. In the example, the normalized range equals $1\overline{R}+2.60\sigma_R$ which falls between the upper warning level, $\overline{R}+2\sigma_R$, and the upper control limit, $\overline{R}+3\sigma_R$. The precision of the results may be suspect.

The normalized deviation is calculated (sample calculation d) by computing the deviation and the standard error of the mean, σ_{m} . The normalized deviation allows one to readily measure central tendency (accuracy) through the use of control charts. Trends in analytical accuracy can be determined in this manner. For this example, the normalized deviation is +0.693 which falls between the upper and lower warning levels. The accuracy of the data is acceptable.

Finally, the experimental error of all laboratories, the grand average, and the normalized deviation from the grand average are calculated in order to ascertain the performance of all the laboratories as a group. Any bias in methodology or instrumentation may be found from these results. Since the results of only one laboratory are listed in the Appendix, the equations are shown without any sample calculations.

Sample Calculations

a. Analysis Data:

Known value = 62 pCi/1

Standard deviation (single determination) = 5 pCi/l

Sample No.	Activity pCi/l
1 2	50 60
3	70

Number of samples (N) = 3

$$\sum_{i} x_{i} = 180 \text{ pCi/1}$$

$$\overline{X} = 60 \text{ pCi/1}$$

b. Experimental Standard Deviation (S).

$$S = \sqrt{\frac{\sum (x_i^2) - \frac{(\sum x_i)^2}{N}}{N-1}}$$

$$S = \sqrt{\frac{50^2 + 60^2 + 70^2 - \frac{(50 + 60 + 70)^2}{3}}{2}}$$

$$S = 10 \text{ pCi/l}$$

- c. Calculation of Normalized Range
 - Step 1. Mean Range (\bar{R})

$$\bar{R}$$
 = $d_2\sigma$ Where d_2 = Central line factor* \bar{R} = (1.693)(5 pCi/l) d_2 = 1.693* for N = 3 \bar{R} = 8.465 pCi/l σ = Standard deviation

(single determination)

Step 2. Control Limit (CL)

CL =
$$\overline{R}$$
 + $3\sigma_R$ Where D_4 = Control Limit factor*
= $D_4\overline{R}$ D_4 = 2.575
= (2.575)(8.465 pCi/1)
= 21.797 pCi/1

Step 3. Standard Error of the Range (σ_R)

$$\sigma_{R} = 1/3 \ (\bar{R} + 3\sigma_{R} - \bar{R})$$

$$= 1/3 \ (D_{4}\bar{R} - \bar{R})$$

$$= \frac{21.797 \ \text{pCi/1} - 8.465 \ \text{pCi/1}}{3}$$

$$= 4.444 \ \text{pCi/1}$$

* Rosenstein, M. and A. S. Goldin, Statistical Techniques for Quality Control of Environmental Radioassay, AQCS Report Stat-1. U.S. Department of Health, Education and Welfare, PHS, Nov. 1964 Step 4. Normalized Range (Range expressed in terms of \overline{R} and σ_R)

Where w = # of \overline{R} and x = # of σ_R defining RIf $R > \overline{R}$ let w = 1 and solve for xIf $R \le \overline{R}$ let x = 0 and solve for wSince, in this example, $R > \overline{R}$ (i.e., 20 > 8.465)

let w = 1 and solve for x $R = w\overline{R} + x\sigma_R$ $x = \frac{R - w\overline{R}}{\sigma_R}$ $x = \frac{20 - (1)(8.465)}{4.444}$ x = 2.598

- : Normalized Range = $1\bar{R} + 2.598\sigma_{R}$
- d. Calculation of the Normalized Deviation
 - 1. Deviation = True Value \overline{X} = 62 pCi/1 - 60 pCi/1 = 2 pCi/1

2. Standard Error of the Mean (σ_m)

$$\sigma_{\rm m} = \frac{\sigma}{\sqrt{N}}$$

$$\sigma_{\rm m} = \frac{5 \text{ pCi/1}}{\sqrt{3}}$$

$$\sigma_{\rm m}$$
 = 2.887 pCi/1

- 3. Normalized Deviation = $\frac{\text{Deviation}}{\sigma_{\text{m}}}$ = $\frac{2 \text{ pCi/l}}{2.887 \text{ pCi/l}}$ = 0.693
- e. Experimental Error = $\frac{\sum (x_i^2) \frac{(\sum x_i)^2}{N}}{N-1}$

Where $\mathbf{x_i}$ are the "1, 2, 3, ...i" results and N equals the number of results in the calculation.

- f. Grand Average = $\frac{\sum x_i}{N}$ where x_i and N are defined as in "e" above.
- g. The Normalized Deviation from the Grand Average is calculated as in "d" except the Grand Average value is used instead of the Known Value.



REQUEST TO PARTICIPATE IN LABORATORY INTERCOMPARISON STUDIES PROGRAM

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REQUEST TO PARTICIPATE IN LABORATORY INTERCOMMENTS STUDIES PROGRAM

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TECHNICAL REPORT DATA (Please read Instructions on the reverse before con	npleting)
1 FEPA-680/4-73-001-b 2	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE	December 1973 (preparation)
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7. AUTHOR(S) Quality Assurance Branch Technical Support Laboratory	8. PERFORMING ORGANIZATION REPORT NO. n/a
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12. SPONSORING AGENCY NAME AND ADDRESS Office of Research and Development U.S. Environmental Protection Agency Washington, DC 20460	13. TYPE OF REPORT AND PERIOD COVERED interim (1973–1974) 14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

The U.S. Environmental Protection Agency's intercomparison studies program for laboratories involved in environmental radiation measurements is described. The types of environmental samples distributed, the analysis required for each sample, the distribution schedule, and the statistical analysis and reporting of results are discussed. Instructions and application forms are included for laboratories desiring to participate in the program.

This document is not a research report. It is designed for use by laboratories participating or desiring to participate in this quality assurance program.

17. KEY WORDS AND DOCUMENT ANALYSIS		
. DESCRIPTORS	b.identifiers/open ended terms	c. COSATI Field/Group
quality assurance quality control quantitative analysis radioactivity statistical quality control		07 05/14 04
Release unlimited (NERC-LV, NTIS)	19. SECURITY CLASS (This Report) Unclassified 20. SECURITY CLASS (This page)	21. NO. OF PAGES 27

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